

This Page Is Inserted by IFW Operations  
and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

**IMAGES ARE BEST AVAILABLE COPY.**

As rescanning documents *will not* correct images,  
please do not report the images to the  
Image Problem Mailbox.

**(12)**

**EUROPEAN PATENT APPLICATION**

**(21)** Application number: 85305480.9

**(51)** Int. Cl.<sup>4</sup>: B 65 D 81/32

**(22)** Date of filing: 31.07.85

**(30)** Priority: 31.07.84 US 635380

**(43)** Date of publication of application:  
05.02.86 Bulletin 86/6

**(64)** Designated Contracting States:  
DE FR GB

**(71)** Applicant: LOCTITE CORPORATION  
708 North Mountain Road  
Newington, Connecticut 06111(US)

**(72)** Inventor: Dunn, David John  
11335 Heritage Drive  
Twinsburg Ohio 44087(US)

**(74)** Representative: Marchant, James Ian et al,  
Elkington and Fife High Holborn House 52/54 High  
Holborn  
London WC1V 6SH(GB)

**(54)** Container and dispenser for a two-component product.

**(57)** An improved container for storing and dispensing a unit dose of liquid curable material and an accelerant therefor, comprising, an inner sealed frangible ampule containing the curable material, an outer flexible sleeve containing the ampule, having a dispensing orifice at one end and being sealed at the other, and fibrous material impregnated with the accelerant, positioned at least partially around the body portion of the ampule closest the orifice. In use, the outer sleeve is compressed to break the ampule, and further pressure causes the liquid curable material to flow past the fibrous material, entraining the accelerant in a controlled amount before exiting through the orifice.

1     Container and Dispenser for a Two-Component Product

---

5         This invention relates to a combined container  
and dispenser for a two component product particularly  
for relatively small amounts of curable liquids which  
require a second component to accelerate the cure by  
intimate mixture therewith. More particularly, it  
relates to such dispensers which are disposable and  
10     contain approximately a unit dose or amount of primary  
material for a single application, such as the adhering  
or sealing of parts to each other.

So-called "unit dose" disposable packaging of liquids  
is known in some industries, for example, the medical  
15     industry, in which single application amounts of a  
liquid to be dispensed are contained in a sealed,  
rupturable ampule further contained within a sealed  
flexible dispenser. The dispenser may be squeezed to  
rupture the ampule and the liquid forced out of the  
20     dispensing orifice by further pressure. These types of  
packages have the advantage of allowing the user to  
quickly apply a relatively exact amount of liquid, with  
no leftover material to dispose of, or waste, or prevent  
against contamination. While on a unit amount basis the  
25     cost is generally higher than for packages of greater  
content, the elimination of waste and over-purchasing  
usually overrides, and the effective cost to the user  
is lower.

This concept has been extended to the dispensing  
30     of liquid adhesives, such as cyanoacrylate adhesives.

Morane et al., U.S. Patent 3,964,643, June 22,  
1976, discloses multi-component container/dispensers  
in which one or more components, which are to be kept  
separate from yet another component, are contained in a  
35     frangible tube held within a flexible outer jacket. The

1 jacket has a rigid portion positioned to exert a  
fracturing pressure on the tube when the jacket is flexed  
or deformed. The other component, apparently a liquid,  
is contained within the jacket, but outside the tube. As  
5 described and illustrated, the dispenser is much larger  
than the unit dose containers just discussed. In fact,  
the outer jacket must have a relatively rigid portion  
positioned proximate the frangible part of the inner  
tube for the purpose of exerting a rupturing pressure  
10 on the tube. It is also noted that, at column 4, two-  
component adhesives are a possible application of the  
described containers.

It is just such an application which is contemplated  
herein, particularly where the relative difference  
15 between the needed amounts of the two-components is  
quite large. For example, there are adhesive systems  
including anaerobic adhesives, acrylic or toughened  
acrylic adhesives, liquid epoxies, and cyanoacrylate  
adhesives, and the like, in which a liquid adhesive base  
20 formulation comprises a first component, and a second  
component which may be used in very small amounts, may  
be combined with the first component to greatly speed  
up the cure time of the adhesive material itself. Such  
second components are variously referred to as accelerators  
25 primers or catalysts. After cure of the base adhesive  
formulation has been initiated, these materials, simply  
stated, accelerate the cure.

Of course once combined with the accelerator,  
such an adhesive has a very short holding time and  
30 must be used relatively quickly. Therefore, when  
relatively large amounts are to be used, the base  
adhesive is packaged separately from the accelerator,  
and the two are normally combined in situ on a part  
or parts to be joined.

35 However, there are a number of applications in

1     which a small total amount, such as a drop or several  
drops, will suffice. In these situations, it would  
be quite advantageous to have a unit dose container  
and dispenser in one, in which the two components may  
5     be kept separate until use, and in use may be swiftly  
combined and dispensed.

It has been found that the configurations and  
teachings of the Morane et al. patent, discussed above,  
are inadequate for a unit dose two-component product.  
10    It is particularly desirable to combine the accelerator  
with the adhesive uniformly, as the adhesive is exiting  
the dispenser orifice, or as close to it as possible.  
Otherwise, a two-component mixture, left in for example  
the relatively large jacket disclosed in Morane et al.,  
15    will start to form gel pockets as the accelerator  
"kicks over" the adhesive. Thus it will not be advan-  
tageous to have the second component completely surround-  
ing the ampule, or tube, containing the first. Addition-  
ally, for the adhesive systems mentioned above, the  
20    liquid accelerators frequently do not mix well with  
liquid adhesives. Again having one liquid surrounding  
the ampule, or tube, with the other is not desirable  
for this reason as well.

It is a principal object of the invention to  
25    provide a combined container and dispenser for a  
two-component product in which at least one component  
is a liquid, in which the two components are kept  
separate until use, and in which the components may  
be uniformly mixed upon usage as the liquid is forced  
30    from the container through the orifice thereof. It  
is a further object to provide such a dispenser in  
single-dose size containing small amounts of liquid  
adhesive and an accelerator thereof.

The present invention provides a combined container  
35    and dispenser of liquid material comprising two-  
components which are to be kept separate within the containe

1     until use and thereafter mixed upon dispensing, the  
container and dispenser comprising an outer flexible  
sleeve having a dispensing orifice at one end and a  
5     seal at the other end, and an inner sealed frangible  
ampule within the sleeve, the ampule containing a  
first liquid component, characterised in that a  
fibrous material within the sleeve is positioned  
between the dispensing orifice and the body portion of  
the ampule closest to the dispensing orifice, the  
10    fibrous material containing on or within its fibers  
a sound component of the liquid to be dispensed. The  
fibrous material may also be positioned at least partially  
around the body portion of the ampule closest to the  
dispensing orifice. When ready for use, the outer sleeve  
15    is compressed sufficiently to break the ampule, and  
further pressure causes the liquid curable material to  
flow through and past the fibrous material, thus  
becoming combined with the accelerant just before  
exiting the orifice.

20           This invention will be described with reference  
to two-component liquid adhesive systems in which  
the speed of cure of the liquid adhesive is accelerated  
by the presence of relatively small amounts of an  
accelerator, catalyst or primer. As before indicated  
25    a number of these types of adhesive systems are well  
known in the literature. For example, anaerobic  
adhesives, such as disclosed in U.S. Patents 3,425,988  
and 3,591,438, and in many other references, are well  
known to be accelerated in cure by the presence of  
30    a transition metal-containing compound, such as a  
copper or iron salt. Other accelerators of anaerobic  
cure are known as well. A growingly familiar class  
of adhesives known as tough acrylics, which are formu-  
lated with elastomeric or rubber-type additives, are  
35    generally two-component systems, in which the activating

1 or accelerating component is comprised of an aldehyde-  
amine condensation product, a preferred species of  
which is a butyraldehyde-aniline material, commercially  
5 sold by Uniroyal under the name Beutene, or by DuPont  
under the name DuPont 808. See for example European  
Patent Publications nos. 0,034,046 and 0,044,166.  
Although cyanoacrylate adhesives, known for fast and  
strong bonding, are normally one-component materials,  
accelerators of cyanoacrylate cure are known and thus  
10 these also may be formulated as two-component systems.  
Of course, epoxy-based systems are typically formulated  
as two-component systems in which the hardener component  
is kept separate from the epoxy resin until use. Liquid  
epoxy systems are known and thus those materials as  
15 well may be advantageously packaged in the dispenser  
of this invention.

Essentially, the dispenser of this invention  
may be used with any chemical formulation or adhesive  
formulation in which an activating component is to  
20 be kept separate from the primary material until use.

Reference is now made to the drawing which is  
a side elevational view of a preferred embodiment of  
the dispenser of this invention. The body of the  
dispenser is cylindrical, although as will become obvious  
25 from the further description, the exact geometric shape  
is not of the essence of the invention. An outer sleeve  
1, comprised of a flexible or compressible material,  
completely contains an ampule or tube 2, which in turn  
contains a quantity of the base liquid material 3,  
30 for example a liquid adhesive material. Ampule 2 is  
completely sealed by any suitable process, and may  
be comprised of any frangible or rupturable material  
which is compatible with the liquid material 3. Typi-  
cally, ampules of this type are thin walled glass struc-  
35 tures, but may obviously be composed of other materials

1 such as plastics. In the illustration of the drawing,  
the diameter of the ampule is smaller than the diameter  
of the sleeve, but preferably fits relatively snugly  
inside the sleeve. While the ratio of the respective  
5 diameters is not critical, a preferred ratio is about  
0.8 to 0.9.

Again referring to the drawing, sleeve 1 possesses  
or is attached to, an orifice or nozzle assembly 4 for  
dispensing purposes. The nozzle assembly 4 and sleeve 1  
10 may be of a unitary construction or may be initially  
separate and sealingly joined by any suitable method.  
In the illustration, and in a currently preferred  
embodiment, the nozzle assembly and sleeve are initially  
separate and are then subsequently seated together to  
15 form a seal through which the liquid material may  
ultimately pass out through the nozzle.

After the ampule 2 is inserted in sleeve 1, the  
other end of the sleeve is sealed by any suitable means  
or material. A preferred method is to pressure inject  
20 a hot melt adhesive material into the sleeve approxi-  
mately touching the other end of the ampule 2 to form  
seal 5.

The distinguishing feature of the dispenser/container  
of this invention is the presence of fibrous material 6  
25 between the nozzle assembly 4 and the end of the ampule  
2 nearest the nozzle assembly. This material 6 can  
be formed of any absorbent or retentive material  
such as gauze, fiber glass, or other suitable fabric  
material. In practice, the material is dipped into  
30 or otherwise has coated on it a predetermined appropriate  
amount of the activating or accelerating second compo-  
nent. Before the ampule 2 is inserted into sleeve 1,  
fibrous material 6 is positioned in the sleeve at



1 the nozzle end thereof such that when the ampule  
is placed therein, fibrous material 6 will be  
positioned between the nozzle assembly and the  
forward end of the ampule.

5 In this manner, when the ampule is broken by  
compression of the sleeve, the liquid material will be  
caused to flow around and through the fibrous material  
on its path to the nozzle, thereby entraining sufficient  
accelerant or activator to speed up the cure of the  
10 liquid material when placed on the part or parts to  
be joined.

The amount of fibrous material 6 to be used and  
the amount of activator or second component material  
to be contained in it is a matter of choice depending  
15 upon the exact two-component system to be dispensed.  
In a preferred aspect, at least some of the body portion  
of the ampule nearest the nozzle assembly will be  
in contact with and surrounded by the fibrous material  
to ensure maximum contact between the liquid in the  
20 ampule and the material itself. However, as before  
stated, it is not desirable or advantageous for the  
fibrous material to completely surround the ampule.  
Thus, for purposes of this invention, the fibrous  
material 6 must be positioned between the nozzle assem-  
25 bly and orifice 4 and the body portion of the ampule  
nearest same such that a majority of the liquid compo-  
nent 3 within the ampule 2 will be caused to pass  
through the fiber 6 upon rupturing the ampule and  
upon dispensing pressure being applied to sleeve 1.

30 The dimensions and materials of construction  
of the inventive dispensers are not critical per se  
to the invention. The dimensions will be largely  
dependent upon the amount of material to be dispensed  
and what amount constitutes a unit dose. For example,  
35 in the case of the unit dose anaerobic adhesive example  
to be discussed below, the ampule itself will have

1 an outer diameter of approximately 6.5 mm, and the  
flexible sleeve length is approximately 5.45 cm. For  
best results in terms of combining the two components  
as stated above, it is preferred that the ampule make  
5 loose frictional contact with the inside of the sleeve;  
in other words, the diameters of the ampule and sleeve  
should be such that the ampule may be easily inserted  
therein but without allowing much side to side movement  
thereafter.

10 The frangible ampule 2 is preferably comprised  
of a thin-walled glass, the composition of which obvi-  
ously should not contain any components which would  
prematurely cause a curing reaction in the liquid  
component 3. For most liquid adhesive applications,  
15 it has been found that boro-silicate glasses are ade-  
quate. Compatible frangible plastics may be used as  
well. The ampule wall thickness, again while not criti-  
cal, should be great enough to withstand normal handl-  
ing pressures, but thin enough to be readily rupturable  
20 upon use. Using the ampule and sleeve dimensions  
mentioned above, a useful ampule will have wall thick-  
nesses of between about 0.10 and 0.38 mm (4 and 15 mils).

For cyanoacrylate adhesives it is highly recom-  
mended to use an ampule container formed from a  
25 borosilicate glass composition, and particularly a glass  
composition in which the boron content ranges from  
about 3.10 to about 3.75% by weight of the glass since  
such a glass has an unexpectedly profound beneficial  
effect on the stability of the alpha-cyanoacrylate  
30 adhesive composition stored therein. Thus, as will  
be hereinafter demonstrated, alpha-cyanoacrylate adhesive  
compositions packaged in borosilicate glass containers  
undergo minimal viscosity changes when stored at temper-  
atures of 23° to 82°C for more than 500 days whereas  
35 the same adhesive when stored in soda-lime glass

1 containers undergo substantial viscosity changes when  
stored at these same temperatures and time intervals,  
and undergo even greater changes in viscosity when  
stored in plastic containers.

5 Borosilicate glasses which have been found to be  
advantageous to prepare the frangible glass containers  
used in the practice of the present invention generally  
contain about 70 to about 73% by weight  $\text{SiO}_2$ , 10 to about  
12% by weight  $\text{B}_2\text{O}_3$ , and 15 to about 20% by weight of  
10 aluminium, alkali and alkaline earth metal oxides such  
as  $\text{Al}_2\text{O}_3$ ,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ ,  $\text{BaO}$  and  $\text{CaO}$ . Borosilicate glasses  
found to be preferred in the practice of the present  
invention have the following composition range:

15	$\text{B}_2\text{O}_3$	about 10 to about 12% by weight
	$\text{SiO}_2$	about 70 to about 72% by weight
	$\text{Al}_2\text{O}_3$	about 6 to about 8% by weight
	$\text{Na}_2\text{O}$	about 5 to about 7% by weight
	$\text{K}_2\text{O}$	about 1 to about 3% by weight
20	$\text{BaO}$	about 0 to about 3% by weight
	$\text{CaO}$	about 0 to about -2% by weight

Sleeve 1 is comprised of flexible material, and  
as indicated before, must be compressible enough to  
25 assist in repturing of the ampule 2 under finger-  
pressure, but must be sturdy enough to withstand normal  
handling, and can be composed of any suitable material  
with any suitable wall thickness to possess these  
characteristics. Continuing with the example of the  
30 dimensions just discussed, sleeve wall thickness  
may usefully range from about 0.13 to 1.27 mm (5 to  
50 mils) depending on the material of construction.  
A preferred material is high or low density polyethylene,  
and with that material the wall thickness may usefully  
35 range from about 0.64 to 1.14 mm (25 to 45 mils). Other

1 materials of construction for the sleeve 1 will be  
apparent to those skilled in the art, such as, for  
example, Nylon, polyethylene-terephthalate, poly-  
acrylonitrile, polyvinylidene fluoride, and poly-  
5 propylene, and the like.

EXAMPLE

An anaerobically-curable adhesive formulation  
is prepared comprising approximately, in percentages  
10 by weight, 60% hydroxypropyl methacrylate, 38.2%  
urethane-methacrylate resin, 0.245% of a 95:5 mixture  
of polyethyleneglycol dimethacrylate and naphtho-  
quinone, 0.49% saccharin, 0.08% acetylphenyl hydrazine,  
0.2% paracresol, 0.49% cumene hydroperoxide, and the  
15 remainder a mixture of metal chelator and methacrylate  
resin. As is well known, anaerobically-curable adhesives  
cure rapidly upon the exclusion of oxygen from the  
system, are generally initiated by the presence of  
hydroperoxy-components, and are capable of being accel-  
20 erated in cure by the presence of transition metal  
ions. The adhesive formulation just described is  
then placed in a borosilicate-glass ampule described  
above which is initially open at one end, after the  
ampule is flushed with dry nitrogen gas. The open  
25 end of the ampule is then immediately, after filling,  
preheated and sealed using a gas-oxygen flame where  
the open end is drawn off to seal the ampule under  
ambient atmospheric conditions. A small circular  
piece of gauze having a diameter of 21 mm is then dipped  
30 in a 15% solution of 12% copper naphthenate in mineral  
spirits. The gauze is permitted to dry and is then  
inserted in a low density polyethylene sleeve having  
the dimensions described above and having a nozzle  
assembly sealingly attached to one end. The gauze  
35 is positioned adjacent the orifice of the nozzle

1 assembly and is then flattened out against the walls  
of the sleeve approximate thereto. The filled ampule  
is then inserted into the sleeve as far forward as  
possible such that the gauze surrounds the forward  
5 end of the ampule and approximately 20% of the sides  
of the ampule.

A white hot melt adhesive sold under the trademark  
Swift 84001 is pressure injected into the sleeve into  
virtual contact with the other end of the ampule.  
10 thus sealing the sleeve at the end opposite the nozzle  
assembly. The hot melt is conveniently applied using  
a Nordson model HM18 system, and is dispensed at a  
temperature of about 135°C (275°F) under approximately  
34.5 to 69 kPa (5 to 10 psi) pressure.

15 To dispense the adhesive, finger pressure is  
applied approximately halfway up the sleeve contain-  
ing the ampule until the ampule is broken. When the  
dispenser is inverted such that the nozzle is pointing  
downward, it will be observed that the bulk of the  
20 liquid anaerobic adhesive flows down the sides of  
the sleeve toward the nozzle, around and through the  
gauze containing the copper salt accelerator, and  
continued finger pressure will force the adhesive  
through the gauze and out through the nozzle. When  
25 applied to parts to be joined such as metal to metal  
parts, or metal to glass, and the parts mated to exclude  
air, fixture occurs typically within 30 to 60 seconds.  
Without the copper salt accelerator present in the  
dispenser, fixture could take as long as 60 minutes.

30 After breaking of the ampule, if the dispenser  
is oriented such that the nozzle is pointing upward,  
it will be seen that the adhesive is pulled away from  
the gauze containing the copper salt accelerator,  
and therefore has much less tendency to prematurely  
35 gel than would be the case if the accelerator component

1 completely surrounded the ampule. Therefore, even  
after rupture of the ampule, the user has much greater  
flexibility in terms of time to dispense the adhesive  
without fear of premature polymerization.

5 As is obvious from the above description, the  
container/dispenser of this invention has broad appli-  
cability for two-component liquid systems which rely  
for their effectiveness on the rapid and uniform mixture  
of the two components, and where it is desirable that  
10 the mixture occur essentially when the adhesive is  
being dispensed. While the invention has been described  
with respect to two-component adhesive formulations,  
it is possible that the dispenser will be useful for  
other two-component liquid systems. Several are men-  
15 tioned in the Morane et al. reference discussed above.  
Others will be obvious from those skilled in the art  
of dispensing liquids. All of these systems are con-  
sidered to be a matter of choice and are deemed to  
be embraced by the concept of the invention just described.

20

25

30

35

1     CLAIMS

1.     A combined container and dispenser of liquid  
material comprising two-components which are  
to be kept separate within the container until  
5     use and thereafter mixed upon dispensing, the  
container and dispenser comprising an outer  
flexible sleeve having a dispensing orifice at  
one end and a seal at the other end, and an  
inner sealed frangible ampule within the sleeve,  
10    the ampule containing a first liquid component  
characterised in that a fibrous material within  
the sleeve is positioned between the dispensing  
orifice and the body portion of the ampule  
closest to the dispensing orifice, the fibrous  
15    material containing on or within its fibres a  
second component of the liquid to be dispensed.
2.     A container and dispenser as claimed in claim 1  
characterised in that the fibrous material is  
20    also positioned at least partially around the  
body portion of the ampule closest to the  
dispensing orifice.
3.     A container and dispenser as claimed in claim 1  
25    or 2 characterised in that the ampule is  
rupturable by the application of finger pressure  
upon the flexible sleeve.
4.     A container and dispenser as claimed in any of  
30    claims 1 to 3 characterised in that the second  
component is an activator for the liquid contained  
within the ampule.
5.     A dispenser and container as claimed in claim 4  
35    characterised in that the liquid in the ampule

1            is an adhesive material and in that the second  
             component is an accelerator of cure of the  
             adhesive.

5           6.    A dispenser and container as claimed in claim 5  
             characterised in that the ampule consists of a  
             glassy material and in that the sleeve is a  
             flexible plastic.

10

15

20

25

30

35



